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## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(21) International Application Number:</b> PCT/US99/26219 <b>(22) International Filing Date:</b> 5 November 1999 (05.11.99)  <b>(30) Priority Data:</b> <table border="0"><tr><td>60/107,182</td><td>5 November 1998 (05.11.98)</td><td>US</td></tr><tr><td>60/109,482</td><td>23 November 1998 (23.11.98)</td><td>US</td></tr><tr><td>60/112,977</td><td>17 December 1998 (17.12.98)</td><td>US</td></tr><tr><td>60/123,375</td><td>8 March 1999 (08.03.99)</td><td>US</td></tr></table> <b>(71) Applicant (for all designated States except US):</b> PHENIX BIO-COMPOSITES, LLC [US/US]; 221 Mohr Drive, Mankato, MN 56001 (US).  <b>(72) Inventors; and</b> <b>(75) Inventors/Applicants (for US only):</b> RIEBEL, Michael, J. [US/US]; 110 Deerplace, Mankato, MN 56001 (US). ROOS, Kenneth, D. [US/US]; 824 Fourth Street, P.O. Box 244, Nicollet, MN 56074 (US).  <b>(74) Agent:</b> MUETING, Ann, M.; Mueting, Raasch, Gebhardt, P.A., P.O. Box 581415, Minneapolis, MN 55458-1415 (US).		60/107,182	5 November 1998 (05.11.98)	US	60/109,482	23 November 1998 (23.11.98)	US	60/112,977	17 December 1998 (17.12.98)	US	60/123,375	8 March 1999 (08.03.99)	US	<b>(81) Designated States:</b> AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
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<b>(54) Title:</b> COMPOSITE PANELS MADE OUT OF CEREAL GRAIN STRAW AND AN ACID-CATALYZED RESIN  <b>(57) Abstract</b>  A composite panel that includes a cereal grain straw and an acid-catalyzed resin.														

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**COMPOSITE PANELS MADE OUT OF CEREAL GRAIN STRAW AND AN ACID-CATALYZED RESIN****Statement of Related Applications**

This application claims the benefit of U.S. Provisional Application No.

- 5 60/107,182, filed November 5, 1998, U.S. Provisional Application No.  
60/109,482, filed November 23, 1998, U.S. Provisional Application No.  
60/112,977, filed December 17, 1998, and U.S. Provisional Application No.  
60/123,375, filed March 8, 1999, each of which is incorporated herein by  
reference.

10

**Field of the Invention**

The present invention relates to the use of an acid-catalyzed resin system  
in the manufacture of composite panels made out of cereal grain straws.

15

**Background**

- The use of agricultural residue or "straw" in the production of composite  
panels is known. The first development of these composite panels used wheat  
straw and was originally formulated in Europe over three decades ago. In the  
early 1990's a renewed interest in this technology occurred due to the  
20 uncertainty of the wood supply as wood particleboard was placed on allocation  
during the late 1980's.

- The early developments of such products typically used wheat straw as a  
filler. In the manufacture of cereal grain straw particleboard, an isocyanate resin  
is conventionally used to fuse the particles together. The advent of MDI  
25 (diphenylmethane diisocyanate) resin technology, for example, has allowed the  
use of a 100% cereal grain straw particleboard. Currently, there are commercial  
scale plants that will produce a "wheatboard" using MDI resin technology.

- In contrast, traditional wood-based particleboard uses less expensive  
phenolic and urea formaldehyde resins. These resins currently do not provide  
30 sufficient adhesion characteristics for wheat or other cereal grain straws.  
Although it is not completely understood, this may be due to the high pH and/or

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buffering capacity of the cereal grain straws and/or the presence of a silica layer and/or natural wax on the outer surface of the straw.

MDI is primarily used for agriculture particleboard as it has a very good affinity to bond most materials together including wood, agricultural fibers, and these materials to metal, plastic, or wood. MDI's tenacity to bond to metal, however, is one of the pitfalls of MDI. Thus, expensive and messy release agents must be used to ensure that the MDI does not stick the particleboard furnish to the metal. Also, the cost of MDI results in a finished particleboard panel that is slightly higher in cost than typical wood-based particleboards.

It would therefore be desirable to employ other resin systems that are easier to use.

### Summary of the Invention

A process is described herein that uniquely utilizes an acid-catalyzed resin system with cereal grain straw to manufacture composite panels, preferably, particleboard. As used herein, "composite panel" includes, but is not limited to, fiberboard (e.g., medium or high density), particleboard, and structural panels. These panels can be used in manufacturing a wide variety of products, including laminates.

Significantly, panels of the present invention have properties that are comparable to, or exceed, American National Standards Institute (ANSI) standards for the specific panel types. Advantageously, the processing of these panels in a manufacturing facility is simplified by negating the use of release agents associated with MDI, for example.

It is commonly accepted in the composite industry that unmodified wheat straw particles can only be bonded with an isocyanate-based resin. However, it is demonstrated herein that by employing an acid-catalyzed resin system that includes, for example, a phenolic resin, with wheat straw in the manufacturing of a composite panel, a product that meets or exceeds industry standards is produced. This is particularly true when an isocyanate resin is used in the core of the panel and the acid-catalyzed resin is used in the faces of the panel.

Thus, the present invention is directed to composite panels (e.g., particleboard) made from a cereal grain straw (e.g., wheat straw) and an acid-

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catalyzed resin system, optionally with an isocyanate resin, and methods of producing such products.

In one embodiment, the invention provides a composite panel comprising a cereal grain straw and an acid-catalyzed resin. The panel can include a  
5 homogeneous distribution of the cereal grain straw and the acid-catalyzed resin. Alternatively, it can include a core and two face layers, wherein at least one face layer comprises an acid-catalyzed resin. Although the core and the two face layers can include the same acid-catalyzed resin, typically and preferably, the core includes an isocyanate resin.

10 Preferably, the cereal grain straw is selected from the group consisting of wheat, oat, rice, barley, millet, rye, triticum grasses, prairie grasses, flax, cannola, and combinations thereof. Preferably, the acid-catalyzed resin is prepared from a resin selected from the group consisting of phenol formaldehyde, melamine, melamine formaldehyde, phenol-melamine  
15 formaldehyde, melamine-urea formaldehyde, melamine-urea-phenol formaldehyde, urea formaldehyde, and combinations thereof. Preferably, the acid-catalyzed resin is prepared from an acid selected from the group consisting of formic acid, fumeric acid, an aromatic sulfonic acid, sulfuric acid, and combinations thereof.

20 In a particularly preferred embodiment, the present invention provides a composite panel comprising a core that includes a cereal grain straw and an isocyanate resin, and two face layers that include a cereal grain straw and an acid-catalyzed resin.

The present invention also provides methods for producing composite  
25 panels (e.g., particleboards) of the present invention. A preferred method includes: combining cereal grain straw and at least one acid-catalyzed resin system; and applying sufficient pressure and temperature to yield a pressed composite panel. Preferably, the temperature of pressing comprises a temperature of about 300°F to about 380°F. The pressure used is sufficient to  
30 obtain the desired thickness and density of the panel. Preferably, the density is about 42 pcf to about 52 pcf.

### Detailed Description of Preferred Embodiments

Phenolic resins, such as phenol formaldehyde resins, are the dominant resins used in wood hardboard, oriented strand board, and laminated veneer lumber. These resins provide a strong bond in a dry environment. They also  
5 provide stability in an aqueous environment over a period of several hours. Phenol formaldehyde liquids have an affinity for wood surfaces, and in the presence of heat condense into a three-dimensionally crosslinked network to provide bonding. When these resins are used with cereal grain straws, however, the curing action or crosslinking is compromised to the point where curing is  
10 significantly slower or does not happen.

Such resins (e.g., phenol formaldehyde and urea formaldehyde resins) are typically "alkaline refined" as they are produced with an alkaline catalyst and a molar excess of formaldehyde. A non-pH sensitive resin like MDI, however, bonds cereal grain straw well to form particleboard. Although the inventors do  
15 not wish to be bound by any particular theory, it is believed that the alkaline nature of the resin and the alkaline nature of the cereal grain straw contribute to the low bond strength.

It has been discovered that resins that can be catalyzed with acid to complete the resin's curing process can provide significant bond strength to  
20 cereal grain straw. It is believed that the acid-catalyzed resin overcomes the high pH and/or effects on buffer capacity of the cereal grain straw and allows for sufficient bond strength to occur. This negates the need for isocyanate resins (e.g., MDI), although they can be used in combination with an acid-catalyzed resin to advantage.

25

### Cereal Grain Straw

As used herein, the term "cereal grain straw" includes the stems from a wide variety of grain crops commonly used for food and feed sources. Some examples of such cereal grain straws include, but are not limited to, wheat  
30 (summer, winter, durum, semolina, etc.), oat, rice, barley, millet, rye, the *triticum* genus of cereal grasses, prairie grasses of the plains states, flax, and cannola.

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The outer layer of the stems of such cereal grain straws is known to inhibit their bonding with standard wood-based particleboard resins. This is believed to be due to the presence of silica-containing and/or waxy materials characteristic of the fiber. Such materials can be removed by pressure refining the fiber. Such pressure-refined fibers are typically only used in products such as medium density fiberboard or hardboard and are not typically used in the production of particleboard. This process, however, is undesirable because the pressure refining requires expensive equipment, high amounts of energy that increases costs, and redrying of the fully saturated fiber.

Thus, cereal grain straw that may or may not be pressure-refined can be used in the production of the composite panels of the present invention, although cereal grain straw that is not pressure-refined, but is mechanically refined, is preferred. Such untreated cereal grain straws can be ground to the desired particle size range by a number of methods, including, for example, hammer milling, chopping, knife refining, and grinding. Thus, in production of composite panels according to the present invention, expensive pressure refining techniques are not required because of the use of an acid-catalyzed resin system that allows for the use of a cereal grain straw that does not have the outer layers removed.

Herein, "furnish" refers to cereal grain straw that has been reduced in particle size. Preferably, this refers to ground straw as opposed to pressure-refined straw. The furnish preferably has a particle size range of about 4 Tyler mesh to about 80 Tyler mesh. Different fractions of this particle size range are typically used to form different portions of the composite panels of the present invention. For example, the inner portions or layers of the composite panels preferably use furnish having a major portion that has particle size of about 4 Tyler mesh to about 20 Tyler mesh, whereas the outer portions or layers of the composite panels preferably use furnish having a major portion that has a particle size of about 20 Tyler mesh to about 80 Tyler mesh. As used herein, "particle size" refers to material that passes the larger mesh screen and is retained on the smaller mesh screen.

### Acid-Catalyzed Resin System

As used herein, an "acid-catalyzed resin system" includes a resin (i.e., one or more resins) and an acid catalyst (i.e., one or more acid catalysts) for curing the resin. Such systems are used to prepare composite panels that can include a homogeneous distribution of one or more types of resin and one or more types of cereal grain straw. Alternatively, and preferably, they can be used to form a composite panel that can include one or more types of resin and/or one or more types of cereal grain straw in multiple layers. For example, a composite panel may be formed from one type of resin system present in the core layer and a different type resin system in the outer (i.e., face) layer.

"Resins" useful in the acid-catalyzed resin system include thermosetting resins, which are capable of further curing or crosslinking upon the addition of an acid. Examples include, but are not limited to, "phenolic resins," such as phenol formaldehyde and phenol-melamine formaldehyde, melamine, melamine formaldehyde, melamine-urea formaldehyde, melamine-urea-phenol formaldehyde, urea formaldehyde, or a combination of these resins. Examples of phenolic resins include those available as PMF 9707 (ARC Resins Corp., Longueuil, QC, Canada) and CRC 153 (Capital Resin Corp., Columbus, OH).

Typically, phenol and formaldehyde are combined and shipped as a colloidal aqueous solution with a solids content between about 30% and about 60% by weight. This is used as provided by the resin manufacturer and then is combined (e.g., by blending) the phenol-formaldehyde resin with an acid catalyst. This same process can be used with urea-formaldehyde resin systems, as well as a number of others, but it is believed that a phenol-formaldehyde resin system is preferred due to its increased performance and water resistance compared to other resin systems, e.g., urea-formaldehyde. Particularly preferred is a phenol-melamine formaldehyde resin.

"Acids" useful in the acid-catalyzed resin system include organic acids and mineral acids. Examples include, but are not limited to, formic acid, fumeric acid, sulfuric acid, as well as aromatic sulfonic acids such as benzenesulfonic acid, phenolsulfonic acid, and toluenesulfonic acid. In a preferred embodiment, the acid is formic acid, which can be obtained under the tradename ARC CATALYST 9700 (ARC Resins Corporation). Although not intending to be

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bound by any particular theory, it is believed that the acidic nature of the acid-catalyzed resin system provides an "etching" action on the cereal grain straw and/or the acid changes the pH and/or buffering capacity of the cereal grain straw, which enhances adhesion between the resin and fibers of the cereal grain  
5 straws.

The types and amounts of resin and acid employed in the acid-catalyzed resin system typically depend upon the type of cereal grain straw selected and the final composite panel attributes desired. For example, in one embodiment of the invention using wheat straw, the acid-catalyzed resin system includes a  
10 phenol-melamine-formaldehyde copolymer, such as PMF 9707 (ARC Resins Corp.) and a formic acid catalyst, such as CATALYST 9700 (ARC Resins Corp.). In another embodiment of the invention using wheat straw, the acid-catalyzed resin system includes a slightly acidic phenolic resin, such as CRC-153 (Capital Resin Corp.), and a toluenesulfonic acid/water catalyst, such as  
15 CRC-350 (Capital Resin Corp.).

The type and amount of resin and acid will effect the cure rate of the resin and bond quality of the panel produced. If too high an amount of acid catalyst is used, it will cause the resin to gel very quickly into a cured state before pressure can be applied to provide intimate contact. Therefore, fiber to  
20 fiber bonding is not achieved. If too low an amount of acid catalyst is used, the resin doesn't effectively cure, even under the application of pressure. Typically, a desired rate of cure is within a range of about 10 seconds to about 25 seconds per millimeter of thickness.

Resins employed in the acid-catalyzed resin system can be used in  
25 composite panels of the invention in an amount of about 3 weight percent (wt-%) solids to about 20 wt-% solids, based on the total amount of solids in the oven dry furnish. Acids employed in the acid-catalyzed resin system can be used in an amount of about 0.5 wt-% to about 15 wt-%, based on the total amount of resin used. These ranges may vary, however, but can be readily determined by one  
30 skilled in the art without undue experimentation.

### Composite Panels

A composite panel according to the invention can be homogeneous with respect to types of resins, types of cereal grain straws, and particle size distribution of the furnish. Typically, however, composite panels include layers of materials. For example, the outer (i.e., face) layer(s) of the composite panels preferably include furnish of a smaller particle size than is used in the inner (i.e., core) layer(s). This is advantageous from an aesthetic perspective, however, the panel may be easier to finish and the bending strength may be improved as well. Face layer material preferably has an average particle size of about 30 Tyler mesh to about 40 Tyler mesh, and the core layer material is typically larger, preferably having an average particle size of about 12 Tyler mesh to about 20 Tyler mesh.

In particularly preferred embodiments, at least one (and preferably both) of the face layers of the composite panels include an acid-catalyzed resin as described above (preferably, a phenolic resin), whereas the core includes an isocyanate resin. A wide variety of isocyanate resins can be used. On preferred one is diphenyl methane diisocyanate (polymeric MDI, which is available from ICI polyurethanes under the RUBINATE trademark and from Bayer under the MONDUR trademark). Many other isocyanates are commercially available from BASF and Dow, for example.

In one embodiment of a three layer construction, the core layer includes an MDI resin and the face layers include an acid-catalyzed resin, such as an acid-catalyzed phenol formaldehyde resin. Examples of panel configurations include a weight ratio of face to core to face of about 20:60:20 to about 25:50:25, although any of a wide range of ratios can be used, depending on the desired result. The use of MDI resin in addition to an acid-catalyzed resin typically yields a lower cost of resin for the total composite and provides improvement in strength and surface hardness over that of 100% MDI straw composite boards.

Composite panels of the present invention can have a wide range of thicknesses and densities. Typically, for particleboard, the thickness of a panel is about 0.25 inch to about 1.5 inches, although panels in a wide variety of thicknesses can be made if desired. Typically, panels can be made according to the invention in a wide variety of densities. For example, densities ranging from

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about 35 pounds per cubic foot (pcf) to about 60 pcf can be achieved, with a preferred range of about 42 pcf to about 52 pcf.

The following is an example of physical properties requirements of three typical grades of particleboard, M-2, M-3, and PBU, and compared with the laboratory test panels. It is not intended that the invention be bound by the requirements of these typical grades as they are used as guidelines when developing a product. Physical property requirements are quite often specified by the end user and may vary, hence the many differing grades. The publication referenced is the ANSI A208.1 - 1999 particleboard standard published by the Composite Panel Association at 18928 Premiere Court, Gathersburg, MD 20879-1569.

Grades	Modulus of Elasticity (psi)	Modulus of Rupture (psi)	Internal Bond (psi)	Screw Pull Face (psi)	Screw Pull Edge (psi)
M-3	398900	2393	80	247	225
M-3	326300	2103	65	225	202
PBU	250200	1595	58	N/A	N/A
Test Samples	Modulus of Elasticity (psi)	Modulus of Rupture (psi)	Internal Bond (psi)	Screw Pull Face (psi)	Screw Pull Edge (psi)
5/8"	502000	2560	88	226	299
1/4"	395000	2700	98	N/A	N/A

#### **Preparation of Composite Panels**

The present invention also provides methods for preparing a composite panel as described herein. Typically, the methods involve combining cereal grain straw and at least one acid-catalyzed resin system, and applying sufficient pressure and temperature to yield a pressed composite panel.

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The cereal grain straw typically has an initial moisture content of about 10 wt-% to about 30 wt-%. This is preferably reduced to less than about 10 wt-% and more preferably, to less than about 5 wt-%. For desired results, the moisture content of the furnish upon combination with the acid-catalyzed resin system is preferably about 2 wt-% to about 10 wt-%, and more preferably about 3 wt-% to about 5 wt-%.

There are several ways in which an acid catalyst can be used to bond a cereal grain straw with a resin. The first method is by directly mixing the acid catalyst with the resin. A predetermined amount of resin solids are measured based on the oven-dry weight of the cereal grain straw furnish being used. The appropriate amount of acid is measured out for the amount of liquid resin being used. The acid catalyst is slowly added to the resin under agitation. Once thoroughly mixed the resin is catalyzed by the acid and is ready for application to the cereal grain straw furnish.

Another method of applying an acid-catalyzed resin to cereal grain straw is by the use of an in-line static mixing device. In this method, the resin is pumped through a metering pump at the specified rate as determined by the amount of oven-dry furnish moving through the system into its separate supply line. A second metering pump is used to pump the acid catalyst based upon the amount of liquid resin being pumped into its separate supply line. The two supply lines merge just prior to an in-line static mixer which is engineered to provide quick thorough mixing of the resin and acid catalyst. Once through the static mixer, the resin is acid catalyzed and ready for application to the cereal grain straw furnish.

Yet another method by which an acid-catalyzed resin may be used in the production of cereal grain straw composite panels is by application of the resin and catalyst separately. The phenolic resin is pumped through a metering pump into its own separate supply line based on the oven-dry weight of the furnish going through the blenders. The phenolic is applied to the furnish in the blender. The acid catalyst is pumped through a metering pump to its own supply line based upon the amount of liquid resin being applied. The acid is added separately to the furnish in the blender. The coating of the furnish with the two liquids plus the mechanical rubbing together of the particles during the blending

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process allows for intimate contact of the two and therefore, the resin becomes acid-catalyzed capable of bonding the cereal grain straw furnish.

If a layered panel is desired (i.e., one in which the core layer includes a different blend of materials than the face layers), a mixture is produced of the desired furnish and acid-catalyzed resin system for each distinct layer. For example, one blend can be produced for the core and one for the two face layers. These are laid down into a mat in the desired thickness ranges and subsequently pressed to form a panel.

Once the acid-catalyzed resin and the cereal grain straw furnish are combined, the material is pressed to form a composite panel. By using the proper resin/acid catalyst combination composite panels can be bonded over a wide range of temperatures, e.g., from room temperature to about 500°F, preferably about 250°F to about 380°F, and more preferably, about 300°F to about 380°F, and a wide range of pressures, e.g., from about 250 pounds per square inch (psi) to about 1000 psi, preferably, about 250 psi to about 750 psi, and more preferably, about 400 psi to about 500 psi. Press time can vary over a wide range as well. Typically, the press time is about 3.5 minutes total to about 10 minutes total, with subsequent hot stacking of about 8 hours to about 15 hours or longer.

An example of a preferred press cycle includes the following: (1) start at 550 psi until the desired thickness is obtained; (2) hold at this distance for one minute; (3) reduce pressure to 250 psi for the desired cook time (3.0 minute); and (4) reduce pressure to 0 psi to degas panel over a period of 40 seconds. A total press time of about 4 minutes to about 6 minutes is used followed by hot stacking the panels overnight to complete curing. Subsequently, the panels can be sanded and treated for a desired surface finish.

### Examples

The invention will be further described by reference to the following detailed examples, which are exemplary and not intended to limit the invention. The examples described herein were of laboratory size, typically about 21 inches by about 21 inches. All percentages are weight percentages unless otherwise noted.

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**Example 1**

Loose or string-baled wheat straw was ground in a hammermill. This milled material was then dried in a dryer, such as a flat-belt gas fired dryer, to a desired moisture content of about 2 wt-% to about 4 wt-%. This dried furnish  
5 was then classified through a screener with different mesh sizes. Coarse, larger particles that passed a 6 mesh screen and were retained on a 20 mesh screen were used for the core layer. Finer, smaller particles that passed a 20 mesh screen and were retained on a 80 mesh screen were used for the face layers.

These core and face furnishes were separately batch blended with the  
10 desired resin. The face furnish was blended with ARC PMF 9707 resin. The amount of resin solids added was 7.5% of the amount of dried face furnish. To this was added the ARC CATALYST 9700 catalyst in an amount of 5%, based on the amount of resin used. The core furnish was blended with 4.5% MDI resin. The furnish was blended in a laboratory batch blender (6-foot diameter,  
15 3.5 feet deep) equipped with a Coil spinning disk atomizer (Model EL-4, Coil Ind., Vancouver, British Columbia) with an atomizing speed of 7500 rpm, a resin addition rate of 1 gram/second, and a blender speed of 18 rpm.

Tri-layer matts were formed in a 21-inch by 21-inch box on an aluminum caul plate. A predetermined amount of blended face furnish was spread out by  
20 hand in the box and leveled. A predetermined amount of blended core furnish was spread out by hand on top of the face furnish and leveled. A second predetermined amount of face furnish was spread out by hand on top of the core furnish and leveled. Slight pressure was applied to remove some of the air from the matt. The face to core weight ratio used was 25:50:25.

25 The box was carefully removed and the formed matt placed in a single-opening heated platen press. The press temperature was 350°F and the maximum pressure was 500 psi with a total press time of 6.0 minutes, which included at least about a 30-second degas time, for 5/8-inch panels and 4.0 minutes for 1/4-inch panels. The target panel density was 45 pcf for the 5/8-inch  
30 panels and 48 pcf for the 1/4-inch panels.

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Results

The physical properties of the panes were evaluated according to ASTM D 1037-96A, as referenced in the ANSI A208.1 - 1999 particleboard standard published by the Composite Panel Association at 18928 Premiere Court,

5 Gathersburg, MD 20879-1569. The results are as follows:

1) 5/8-inch panels: Internal bond = 87.5 psi; MOE = 502,000 psi; MOR = 2,560 psi; Screw through face 226 lbs (only could get screw in 1/2 not 2/3), Screw through edge 299 lbs.

2) 1/4-inch panels: Internal bond = 97.7 psi, MOE = 395,000 psi, MOR =  
10 2,700 psi.

All values met or exceeded the ANSI standard for M3 particleboard or in the case of the 1/4-inch panel specifications for underlayment. This experiment indicates the feasibility of manufacturing cereal grain particleboard with MDI bonded cores and acid-catalyzed phenolic resin faces.

15

All patents, patent applications, and publications are incorporated by reference herein as though individually incorporated by reference. Various modifications and alterations of this invention will become apparent to those skilled in the art without departing from the scope and spirit of this invention,  
20 and it should be understood that this invention is not to be unduly limited to the illustrative embodiments set forth herein.

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**What Is Claimed Is:**

1. A composite panel comprising a cereal grain straw and an acid-catalyzed resin.
2. The composite panel of claim 1 comprising a homogeneous distribution of the cereal grain straw and the acid-catalyzed resin.
3. The composite panel of claim 1 comprising a core and two face layers, wherein at least one face layer comprises an acid-catalyzed resin.
4. The composite panel of claim 3 wherein the core and the two face layers comprise the same acid-catalyzed resin.
5. The composite panel of claim 3 wherein the core comprises an isocyanate resin.
6. The composite panel of claim 3 wherein the acid-catalyzed resin of the at least one face layer comprises a phenolic resin.
7. The composite panel of claim 1 comprising a core and two face layers, wherein the core layer comprises cereal grain straw having a mesh size of about 12 to about 20.
8. The composite panel of claim 7 wherein the face layers comprise cereal grain straw having a mesh size of about 30 to about 40.
9. The composite panel of claim 1 wherein the cereal grain straw is selected from the group consisting of wheat, oat, rice, barley, millet, rye, triticum grasses, prairie grasses, flax, cannola, and combinations thereof.
10. The composite panel of claim 1 wherein the acid-catalyzed resin is prepared from a resin selected from the group consisting of phenol formaldehyde, melamine, melamine formaldehyde, phenol-melamine

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formaldehyde, melamine-urea formaldehyde, melamine-urea-phenol formaldehyde, urea formaldehyde, and combinations thereof.

11. The composite panel of claim 10 wherein the acid-catalyzed resin is prepared from an acid selected from the group consisting of formic acid, fumeric acid, an aromatic sulfonic acid, sulfuric acid, and combinations thereof.

12. A composite panel comprising:  
a core comprising a cereal grain straw and an isocyanate resin; and  
two face layers comprising a cereal grain straw and an acid-catalyzed resin.

13. The composite panel of claim 14 wherein the face to core to face weight ratio is about 20:60:20 to about 25:50:25.

14. The composite panel of claim 13 wherein the cereal grain straw is selected from the group consisting of wheat, oat, rice, barley, millet, rye, triticum grasses, prairie grasses, flax, cannola, and combinations thereof.

15. The composite panel of claim 13 wherein the acid-catalyzed resin is prepared from a resin selected from the group consisting of phenol formaldehyde, melamine, melamine formaldehyde, phenol-melamine formaldehyde, melamine-urea formaldehyde, melamine-urea-phenol formaldehyde, urea formaldehyde, and combinations thereof.

16. The composite panel of claim 13 wherein the acid-catalyzed resin is prepared from an acid selected from the group consisting of formic acid, fumeric acid, an aromatic sulfonic acid, sulfuric acid, and combinations thereof.

17. The composite panel of claim 13 which has a density of about 35 pcf to about 60 pcf.

18. A method of preparing a composite panel comprising:

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combining cereal grain straw and at least one acid-catalyzed resin system; and  
applying sufficient pressure and temperature to yield a pressed composite panel.

19. The method of claim 18 wherein the temperature of pressing comprises a temperature of about 300°F to about 380°F.
20. The method of claim 18 wherein the composite panel has a density of about 42 pcf to about 52 pcf.
21. The method of claim 18 wherein the composite panel comprises a homogeneous distribution of the cereal grain straw and the acid-catalyzed resin.
22. The method of claim 18 wherein the composite panel comprises a core and two face layers, wherein at least one face layer comprises an acid-catalyzed resin.
23. The method of claim 22 wherein the composite panel core comprises an isocyanate resin.
24. The method of claim 18 wherein the cereal grain straw is selected from the group consisting of wheat, oat, rice, barley, millet, rye, triticum grasses, prairie grasses, flax, canola, and combinations thereof.
25. The method of claim 18 wherein the acid-catalyzed resin system comprises a resin selected from the group consisting of phenol formaldehyde, melamine, melamine formaldehyde, phenol-melamine formaldehyde, melamine-urea formaldehyde, melamine-urea-phenol formaldehyde, urea formaldehyde, and combinations thereof.

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26. The method of claim 18 wherein the acid-catalyzed resin system comprises an acid selected from the group consisting of formic acid, fumeric acid, an aromatic sulfonic acid, sulfuric acid, and combinations thereof.

# INTERNATIONAL SEARCH REPORT

International Application No  
PCT/US 99/26219

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 B27N3/02 B32B5/16 B32B5/22 B32B9/02

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 B27N B32B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 514 255 A (MAXWELL JOHN W ET AL) 30 April 1985 (1985-04-30)  column 1, line 20 - line 22; claims 1,12-15 column 7, line 16 - line 21 ---	1,2,10, 18,19, 21,25
X	DATABASE WPI Section Ch, Week 199213 Derwent Publications Ltd., London, GB; Class A32, AN 1992-102470 XP002131525 & JP 04 047902 A (KOYO SANGYO CO LTD), 18 February 1992 (1992-02-18) see, in particular, statement in the abstract "in the presence of an (in)organic acid" abstract --- -/-	1-4,6,9, 10,18, 19,21, 22,24,25

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

### \* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

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"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

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# INTERNATIONAL SEARCH REPORT

Inter. Appl. No.

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